OPTIMIZATIONS AND RECYCLING INDUSTRIAL WASTE (PALM OIL FLY ASH) AS A PIGMENT IN COATING TECHNOLOGY

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Especially dedicated to my beloved parent and family members;
Mohd @ Mohd Nor Bin Jusoh and Hamsiah Binti Jaafar;
Syahian & Nurul Haslinda, Mohd Najib & Huhaida, Mohd Shahril & Norfatiha, Ahmad Kamilrul;
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My love, Fatin Syahirah;
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May Allah S.W.T always bless us all.

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ABSTRACT

In this study, the feasibility of palm oil fly ash (POFA) as a pigment in paint technology is investigated. The utilization of POFA as paint pigment is favorable due to its stability and non-toxicity. On the other hand, producing colour pigment by using POFA as pigment element reduces the solid waste in palm oil industry, in addition to the advantages of inexpensiveness and ready access to POFA feedstock. The objective of this research are to identify the optimum composition of POFA in combination of binder, solvent and additives as pigment in paint, to examine the heat resistant behavior of POFA paint and investigating the POFA paint coating ability to withstand from the paint testing. In general, this research hopes to improve the technology of coating and also can be a significant contribution in the academic, research, development and field studies related to the paint technology. The optimum preparation and thermal conductivity of paint that prepared from the waste of palm oil fly ash (POFA) were investigated. For optimum pigment preparations, the aqueous precursor of 45 % wt POFA mixed with 25 % wt sulphur, 10 % wt graphite fine powder and 20 % wt calcium hydroxide was sintered at 700 – 800 °C to produce a complete crystalline product. The sintered product was milled and become grayish fine powder pigment. From the SEM images, the grain size approximately 0.404 µm. The pigment was mixed with binder, solvent, and additives and grinded to become a paint and tested (glossy test, hardness test, adhesion test, and thermal conductivity test). From the result, the optimum composition of a paint are 22 % wt POFA pigment, 29 % wt binder, 39 % wt solvent and 10 % wt additives (cobalt 10%, lead 32% and N.C Solution). It also discovers that the paint is good insulator with thermal conductivity approximately 78.95 W/m.°C.

ABSTRAK

Dalam kajian ini, abu terbang kelapa sawit atau POFA berkemungkinan dijadikan sebagai pigmen dalam teknologi cat disiasat. Penggunaan POFA sebagai pigmen cat adalah baik kerana kestabilan dan bukan ketoksikan bahan tersebut. Sebaliknya, menghasilkan pigmen warna dengan menggunakan POFA sebagai elemen didalam pigmen dapat mengurangkan sisa pepejal dalam industri minyak sawit, sebagai kelebihan tambahan bagi bahan mentah POFA. Objektif kajian ini adalah untuk mengenalpasti komposisi optimum POFA dengan mengabungkan bahan seperti pengikat, pelarut dan bahan tambahan sebagai pigmen dalam cat, untuk mengkaji cat POFA pada daya tahan haba dan menyiasat keupayaan lapisan cat POFA dalam standard ujian cat yang ditetapkan. Secara umum, kajian ini diharap dapat meningkatkan teknologi salutan dan juga boleh dijadikan sebagai sumbangan penting dalam bidang akademik, penyelidikan, pembangunan dan bidang kajian yang berkaitan dengan teknologi cat. Kajian tentang penyediaan optimum dan kekonduksian haba cat yang disediakan daripada sisa abu terbang kelapa sawit (POFA). Bagi penyediaan pigment yang optimum, 45% wt POFA dicampur dengan 25% wt sulphur, 10% wt serbuk grafik halus dan 20% wt kalsium hidroksida telah disinter pada 700-800 °C untuk menghasilkan produk kristal yang lengkap. Produk yang dibakar akan dikisar dan menjadi serbuk pigment kekelabuan yang halus. Dari keputusan imej SEM, saiz butiran adalah lebih kurang 0,404 mikron. Pigmen ini akan dicampurkan dengan pengikat, pelarut, dan bahan tambahan dan dikisar untuk menjadi cat dan diuji degan ujian kilatan, ujian kekerasan, ujian lekatan, dan ujian kekonduksian haba). Dari keputusan itu, komposisi cat yang optimum mempunyai 22% wt POFA pigmen, 29% wt pengikat, 39% wt pelarut dan 10% bahan penambah (cobalt 10%, lead 32% dan N.C Solution). Ia juga mendapati bahawa cat ini adalah penebat yang baik dengan kekonduksian haba kira-kira 78.95 W/m.°C.

CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

POFA	-	*Palm Oil Fly Ash*
SiO_2	-	*Silicon Dioxide*
Al_2O_3	-	*Aluminium Oxide*
Fe_2O_3	-	*Iron Oxide*
SO_3	-	*Sulphur Trioxide*
min	-	*Minimum*
%	-	*Percent*
µ	-	*Micron*
% wt	-	*Weight Percent*
BC	-	*Before Century*
$Ca(OH)_2$	-	*Calcium Hydroxide*
Si	-	*Silicon*
Al	-	*Aluminium*
°C	-	*Degree Celsius*
°C/min	-	*Degree Celsius Per Minute*
h	-	*Hour*
$(CH_3)_2CO$	-	*Acetone*
g	-	*Gram*
SEM	-	*Scanning Electron Microscope*

θ	-	*Theta*
°	-	*Degree*
XRD	-	*X-Ray Powder Diffraction*
ASTM	-	*America Society For Testing And Materials*
H	-	*Hardness*
BS	-	*British Standards*
DIN EN	-	*Deutsches Institut Für Normung (German Institute For Standardization)*
ECCA	-	*European Coil Coating Association*
ISO	-	*International Organization For Standardization*
GU	-	*Gloss Unit*
W	-	*Watt*
W/m.°C	-	*Watts Per Meter Degree Celsius*
K	-	*Kelvin*
$\dot{Q}$	-	*Rate Of Heat Transfer*
k	-	*Thermal Conductivity*
A	-	*Cross-Sectional Area*
T_1-T_2	-	*Temperature Different*
L	-	*Thickness*
EDX	-	*Energy-Dispersive X-Ray*
Be	-	*Beryllium*
C	-	*Carbon*
O_2	-	*Oxygen*
Mg	-	*Magnesium*

S	-	*Sulfur*
K	-	*Potassium*
Ca	-	*Calcium*
Mo	-	*Molybdenum*
SiO_2	-	*Quartz*
$KAL_2(Si_3Al)O_{10}(OH,F)_2$	-	*Muscovite*
$KAlSiO_4$	-	*Kalsilite*
$K_8Al_6Si_6O_{24}S_4$	-	*Lazurite*
cP	-	*Centipoise*

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1. BACKGROUND OF THE STUDY

The manufacturing of paint dates back to the 1700s. It is mainly used for artwork initially due to its high production cost. The technology expands continuously for many years, with techniques being passed down from generation to generation by travelling craftsmen. A few centuries later, through further innovations and improvement, paints factories are opened in Europe and America. The mass production of paint in the nineteenth century marks the starting point of price down; to such an extent that houses start to be painted. Now, in the twentieth century, the chemical composition of paint has been unlocked, indicating that the manufacturing of paint has finally moved from being an art to a science (Waldie, 1983).

Paint is widely used to protect surfaces of objects and for colouring purposes. Its applications range from car paint coating to road marking. Different sorts of paint are used in different kinds of applications. The basics ingredients for most of the paint are pigments, binders, solvents and additives. The protective coating used for corrosion protection consists of pigment too. Despite the apparent purpose of pigmentation on the layer is to provide colour and opacity, correct formulation of pigment into the protective layer is far more than that. For corrosion protective coating, pigment works by providing inhibition or passivation of metal surfaces, preventing corrosion and strengthening the paint film.

The manufacturing of paint is rather simple. The pigment, binder and thinner are blended in the correct proportions, such that the final finished film is continuous, smooth and attractive to the eyes when the paint is applied to the surface. In the paint technology, there are two types of pigment - the organic pigment and inorganic pigment. The organic pigment is for decorative purposes, while the inorganic pigment is for protective purposes (Talbert, 2008). Certain types of pigment increase the heat, abrasion, acid, or alkali resistance of the layers. The particle size and shape of pigment, ease of wettability by the binder, and attributes associated with a particular density contribute to the characteristics of viscosity and wet coating application significantly, in addition to dry lining and protection (Gedeon, 1995).

Various industrial wastes can be used as the pigment in paint technology, where one of them is fly ash. Fly ash, also known as flue-ash, is one of the residues that commonly obtained from the flue gases of furnaces at pulverised coal power plants. When coal is burnt in pulverised coal boilers, the minerals, entrained in the coal, are thermally transformed into chemical species that are reactive or could be chemically activated. On the other hand, bottom ash refers to ash which does not rise. Fly ash is generally captured from the chimneys of coal-fired power plants, whereas bottom ash is removed from the bottom of furnaces.

Palm oil fly ash (POFA) powder is residue obtained from the dust collectors in the boiler that uses palm oil dregs as fuel. The boiler here is usually found in power plant industries. The palm oil residue, palm fibre and shells are burnt in the boiler at about 800˚C to 1000˚C to produce steam for electricity generation in biomass thermal power plants (Science, 2012). Dregs of oil palm, combining with the kernel and its bunch are turned into fly ash after burning in a boiler. Fly ash is the result of burning, and has strong chemical bonding, high strength and low water absorption ability.

1.2. PROBLEM STATEMENT

The relatively high price of the paint in current market is mainly attributed to the cost of its raw material. To reduce the production cost of paint, further study into the use of alternative low cost materials forms the key component in the paint technology nowadays.

Biomass waste can be utilized as the pigment in paint technology through chemical processes. Silicon dioxide that exists in pigment composition, known as extender, is one of the components found in POFA. It thus indicates that POFA has high potential to be a low cost alternative to current raw material used in paint, since it is a type of waste product. The POFA may be used to produce the multifunction paint that is water resistant and heat resistant.

In addition to cost efficiency, utilization of POFA as raw material in paint pigment helps to solve environmental problems, for instance, ground water contamination and spills of storage.

1.3. OBJECTIVES OF RESEARCH

The objectives of this study are summarized as follows:

1) To identify the optimum composition of POFA in a combination of binder, solvent and additives as the pigment in paint.
2) To evaluate the heat resistant characteristic of POFA paint.
3) To investigate the ability of POFA in paint coating using paint standard testing.

1.4. SCOPE OF RESEARCH

The scopes of this study are summarized as follows:

1) The study focused on the standard operation procedure of making paint by using POFA as a pigment.
2) The paint properties were examined according to the composition ratio of paint component and mixing ratio of raw materials in the pigment.
3) The POFA was used as the material in the manufacturing of pigments of paint.

1.5. SIGNIFICANT OF RESEARCH

In general, the research hopes to improve the technology of coating. It can also be a significant contribution to the academic, research, development and field studies related to the paint technology.

The research is important to produce a quality paint through the standard procedure in coating technology. Besides that, fly ash has a high potential to be developed as a pigment in paint application in order to improve the quality of paint. Furthermore, the data collected in this experiment are able to contribute to the knowledge in regards to the uses of biomass waste as one type of pigment in the paint industry.

The research may be able to reduce environmental problems, such as ground water contamination and spills of bulk storage. Besides that, the added value of the industrial waste can be identified through this study.

1.6. AIM OF RESEARCH

The aim of the research is to exploit the potential of industry waste, specifically, POFA, as the pigment used in the paint technology, due to its abundance, inexpensiveness and ready accessibility. The making of paint with the POFA as the pigment is operated in accordance with the standard procedures, and the optimum mixing ratio of raw materials that gives better paint properties has been identified.

1.7. OVERVIEW OF THESIS

The thesis begins with Chapter 1, with an introduction to the history of paint. The needs of the study are discussed, in addition to the significance of the conducted study explained in this thesis. The objectives, scope, and overview of this thesis are then described.

Chapter 2 provides a review of industrial waste, palm oil fly ash (POFA). The review of paint components, including pigment, binder, additives and solvents are presented. The characteristics of the heat resistant paint are reviewed.

Chapter 3 discusses the methodology used to conduct this study. Specifically, the procedures of pigment preparation, paint preparation and characterization of paint properties were discussed in detail.

Chapter 4 presents the analysis results obtained during the paint pigment preparation initially. From the results, the pigment sample with the best quality was used in producing the POFA paint. The obtained optimal paint pigment and the POFA paint sample were discussed in detail in this chapter. Lastly, Chapter 5 discusses the conclusion and recommendation the entire work.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

A literature review is the first step towards considering the critical points of current knowledge (Baglione, 2012). In this chapter, the selected studies of paint and pigment, specifically, patents, research papers, journals, books and available products, were evaluated.

The literature review of composition, paint components and characterization was conducted as well. This is to explore and investigate the related applicable component with practical techniques on the existing paint that could assist in the paint research.

2.2 PALM OIL FLY ASH

Palm oil fly ash (POFA) is the industrial waste formed after the combustion of palm oil dreg. It is an amorphous solid, meaning that it is shapeless or non-crystalline solid. Ash can be classified into two types, namely, the bottom ash and fly ash. The term of "biomass" refers to a material that derived from living or recently living biological organisms. It is also often used to refer to plant material, food processing and preparation, and domestic organic waste that can be formed as the source of biomass. The temperature required to burn oil palm dregs in the boiler is between 700-1000 °C (Tangchirapat, 2007).

On the other hand, the term of "ash" is referring to the non-combustible mineral content of biomass. Fly ash consists of very small particles of ash, and will be carried out through the flue gas. Various types of biomass produce ash having similar pozzolanic activity as coal fly ash, including palm, rice husk, wheat straw, sugar cane straw and wood. Generally, the fly ash is produced from palm oil, which has been blended and crushed and went through the burning tunnel (boiler). Here, palm oil dreg means the bunch and kernel of palm oil which have been pressed by a special press machine. The burning process of palm oil dreg in a furnace is the first step in order to produce fly ash (Tulus, 2011). The burning process makes dreg into small particles. Subsequently, a fan is used to pull out lighter fly ash and leave heavier fly ash in a special container. This process to isolated fly ash and bottom ash. During combustion, bottom ash is the ash left behind in or under the grate or combustion region, or at the bottom of a gasifier. The resultant fly ash in the first stage is produced in the lay bar of the system. This process will be repeated in the second stage but with different sizes of particles (Mustaffa Bin Hj Ibrahim, 2010). In the last stage, fine particles are stored below the chimney boiler. The fan is used to inhale lighter fly ash or granule from the chimney.

POFA is physically powder, fine amorphous and dark grey in colour. The colour of fly ash depends on the percentage of carbon in POFA. The more the carbon in fly ash, the darker the grey colour. In addition, POFA also has a variety of particle sizes and similar forms of spherical. One example of an electron micrograph of POFA is shown in Figure 2.1 (Awal, 2010).

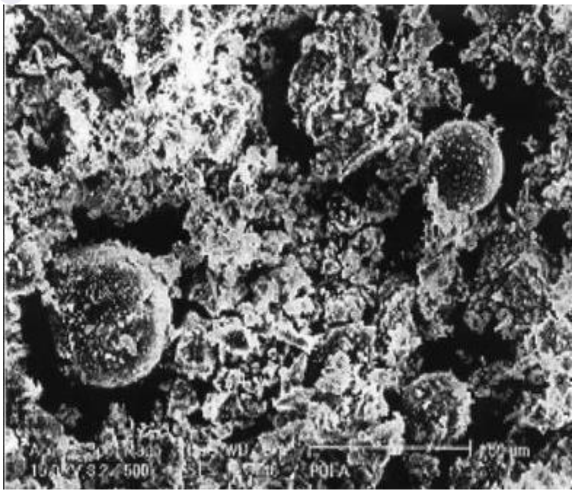

Figure 2.1: Scanning Electron Micrograph from POFA (Awal, 2010)

POFA has its own benefit as compared to other new waste materials used in industries. In addition to its inexpensiveness and abundance, POFA can be utilised in new paint technologies. POFA can be classified into two classes - F and C, based on the chemical composition of the fly ash (Tulus, 2011). The chemical requirements to classify any fly ash in accordance with ASTM C 618 standard are shown in Table 2.1.

Table 2.1: Chemical Requirement for Fly Ash Classification (Awal, 2010)

Properties	**Fly Ash Class**	
	Class F	**Class C**
Silicon Dioxide (SiO_2) + Aluminium Oxide (Al_2O_3) + Iron Oxide (Fe_2O_3), min, %	70.0	50.0
Sulphur Trioxide (SO_3), min, %	5.0	5.0
Moisture content, min, %	3.0	3.0
Loss on ignition	6.0	6.0
The use of class F fly ash containing up to 12% lost ignition may be approved by the user if acceptable performance result are available.		

The chemical compositions of POFA were determined by standard wet chemical analysis method as in (Tulus, 2011). The wet sieve analysis of POFA showed that 78% of the particles were below 4µm size. The chemical analysis of various oxide percent content, (wt %) is shown in Table 2.2 (Tulus, 2011).

Table 2.2: Chemical Composition and Oxide Percent Content (Awal, 2010)

Chemical Compositions	**Oxide Percent Content, (wt %)**
SiO_2	62.12
Al_2O_3	21.30
Fe_2O_3	5.55
TiO_2	1.38
MgO	1.58
CaO	0.53
K_2O	4.24
Loss on ignition	3.3

According to existing studies, the characteristic of thermal insulation has been observed in construction materials contain POFA. The construction materials with a high percentage of POFA possess low thermal conductivity properties and high thermal resistance (Balo, 2013).

LIST OF CONFERENCES

1. 2nd NATIONAL CONFERENCE ON KNOWLEDGE TRANSFER (KTP02) - *Preparation of Palm Oil Fuel Ash Composite as Green Pigment*, 9-11 September 2014, Putrajaya. (Presenter)
2. 5TH INTERNATIONAL CONFERENCE ON MECHANICAL AND MANUFACTURING ENGINEERING 2014 (ICME2014) - *Preparation of Palm Oil Fuel Ash Composite as Green Pigment, Iron Oxide Rust as Raw Material for the Production of Red Pigment in Paint Industry*, 29-30 October 2014, Bandung, Indonesia. (Participant)
3. Research and Innovation Festival 2014, 2 & 3 November 2014, Universiti Tun Hussein Onn Malaysia. (Presenter)
4. Pertandingan Memasyarakatkan Inovasi Pelajar IPT MyINOVASI 2014, 15 & 16 December 2014, Putrajaya. (Presenter)
5. 26th International Invention, Innovation & Technology Exhibition (ITEX15) - *Gloss Paint: Produce from Palm Oil Fly Ash*, 21-23 May 2015, Kuala Lumpur Convention Centre. (Presenter)
6. 3rd International Conference Mechanical Engineering Research (ICMER 2015) – *The Optimum Preparation and The Thermal Conductivity of Novel Glossy Paint Prepared from Waste of Palm Oil Fly Ash, POFA Composite,* 18-19 August 2015, Kuantan. (Participant)
7. Seoul International Invention Fair 2015 (SIIF2015) – *Gloss Paint Produce from Palm Oil Fly Ash (POFA)*, 26-28 November 2015, Seoul, South Korea. (Participant)

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1. Chee Kiong Sia, S. Hakimi Mohd, Pauline Ong, Kuang Jie Fie, 2014. *Iron Oxide Rust as Raw Material for the Production of Red Pigment in Paint Industry*. Applied Mechanics and Materials, vol. 660, pp. 229-233, ©Trans Tech Publications, Switzerland.
2. Chee Kiong Sia, S. Hakimi Mohd Nor, Pauline Ong, Wei Ming Ng, 2014 P*reparation of Palm Oil Fuel Ash Composite as Green Pigment*, Applied Mechanics and Materials, vol. 660, pp. 190-194, ©Trans Tech Publications, Switzerland.
3. C.K. Sia, W. L. Tan, O.L.C. Narong, S. Hakimi Mohd Nor, S. K. Yee and P. Ong, 2015. *Optimum Preparation and Thermal Conductivity of Novel Glossy Paint Prepared from Waste of Palm Oil Fly Ash Composite (SCOPUS),* International Journal of Automotive and Mechanical Engineering (IJAME), ISSN: 2229-8649; 2180-1606, vol. 12, page 3058-3064, ©Universiti Malaysia Pahang.

AWARDS AND ACHIEVEMENTS

1. **Bronze Award** - 26th International Invention, Innovation & Technology Exhibition (ITEX15) - *Gloss Paint: Produce from Palm Oil Fly Ash*, 21-23 May 2015, Kuala Lumpur Convention Centre.
2. **Silver Award** - Seoul International Invention Fair 2015 (SIIF2015) – -, 26-28 November 2015, Seoul.
3. Memorandum of Understanding between UNIVERSITI TUN HUSSEIN ON MALAYSIA and AMPLE GREEN COATING ENTERPRISE SDN BHD on RESEARCH AND DEVELOPMENT ON USAGE OF INDUSTRIAL WASTE AS COATING MATERIAL.
4. New Patent Application. Gloss Paint: Produce from Palm Oil Fly Ash (POFA).
   Reference No: UTHM/PPI/600-5/Jld7 (102)
   IP Reference No: IPR/UTHM/GA/29/2015